

Studying Distribution System Solids: Approach and Application

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Project Objective

- Briefly discuss approach taken by EPA and methods used to study distribution system solids
- Discuss how the information can be used and illustrate with examples

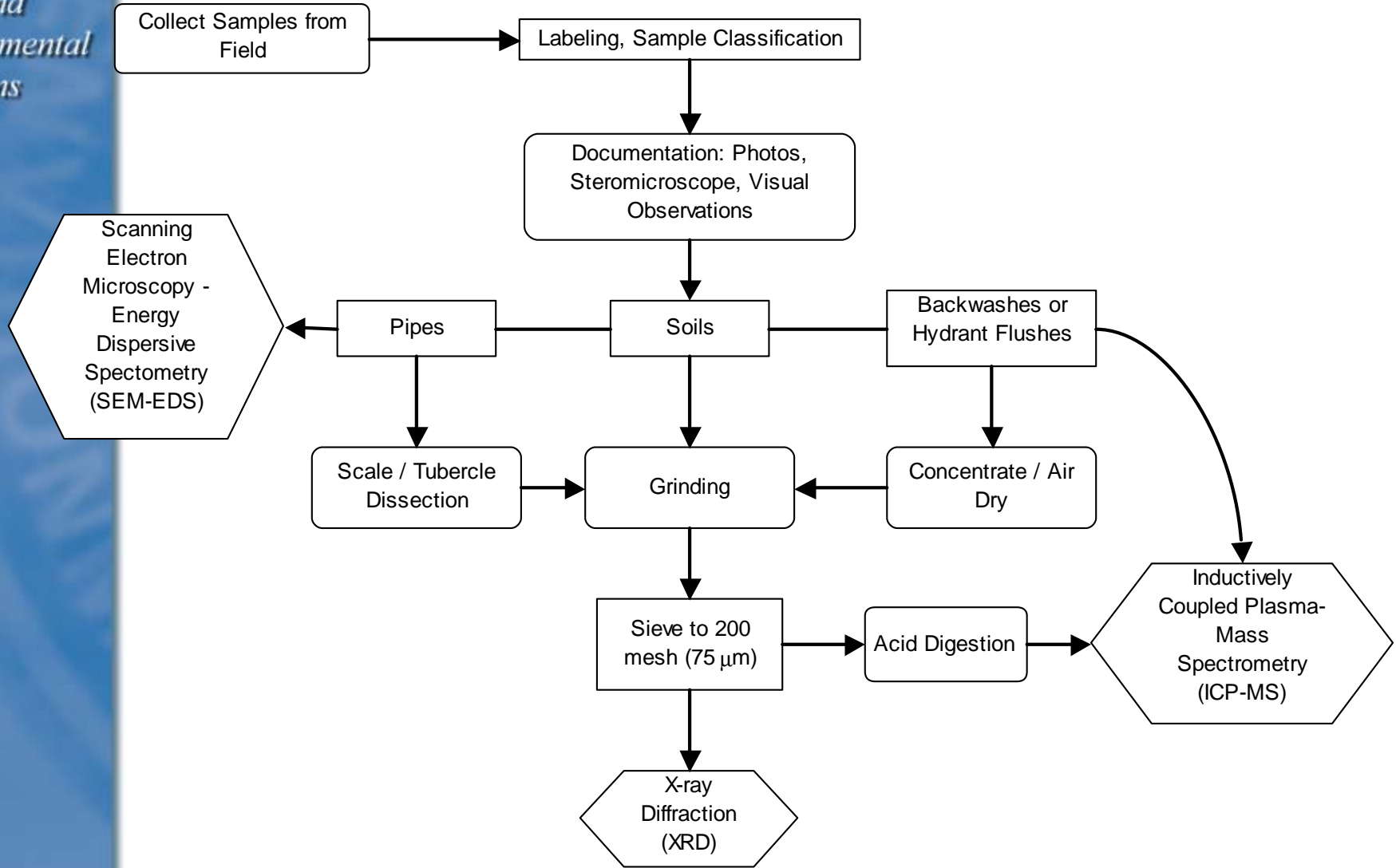
Distribution System Solids

- Corrosion products (CuO , FeOOH , PbCO_3)
- Precipitated solids (CaCO_3 , MnO_2)
- Sediment

Why Study the Characteristics of D.S. Solids?

- Understand corrosion mechanisms
- Trace contaminant accumulation
- Understand red water generation
- Predict metal solubility
- Identify solids of unknown nature
 - Deposited solids
 - Suspended solids

D.S. Solid Sample Preparation Flowchart



Fire Hydrant Flush



Pipe Material



PVC pipe



Iron pipe



Copper Pipe

Analysis Techniques

X-ray Diffraction

- Identification of crystalline minerals
- Crystal size approximation

SEM - Energy Dispersive Spectrometry

- High magnification micrographs
- Elemental composition and mapping

Analysis Techniques

Acid Digestion and ICP – MS

- Provides quantitative elemental composition of solid samples
- This technique is destructive

X-Ray Florescence

Provides semi-quantitative elemental composition without destroying the sample.

Transmission Electron Microscopy

Analysis Techniques

X-ray Photoelectron Spectrometry

Determines oxidation state, bonding energy, bond type, and chemical composition

Electron Microprobe

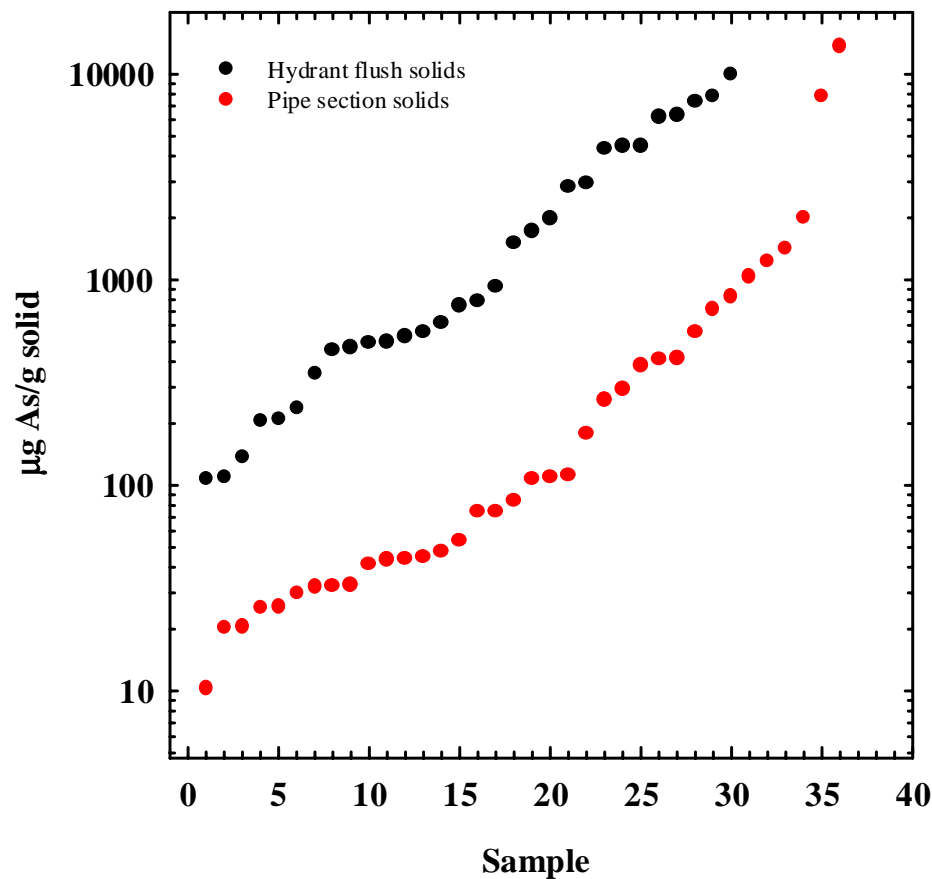
Determines the chemical composition of very small samples, and produce high resolution elemental maps

Analysis Approach

MULTIPLE TECHNIQUE ANALYSIS
APPROACH IS CRITICAL

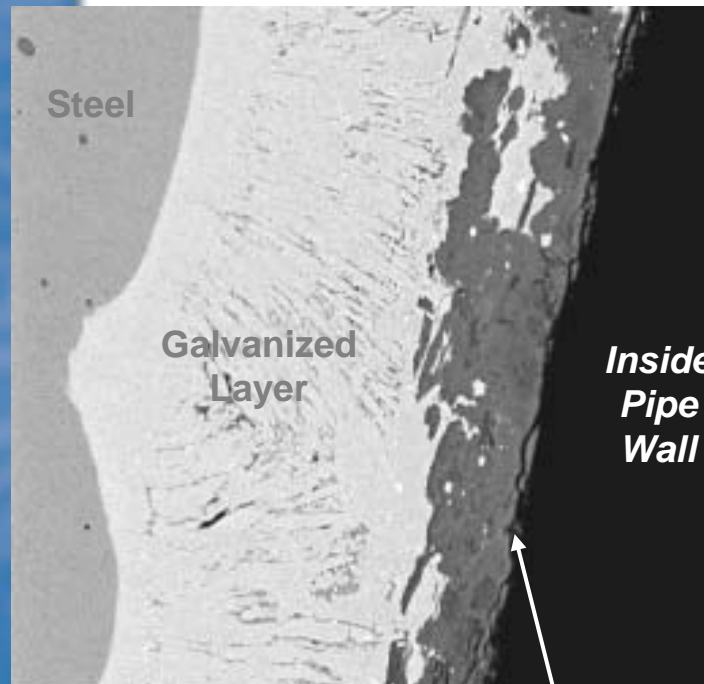
Elemental Composition

ICP-MS

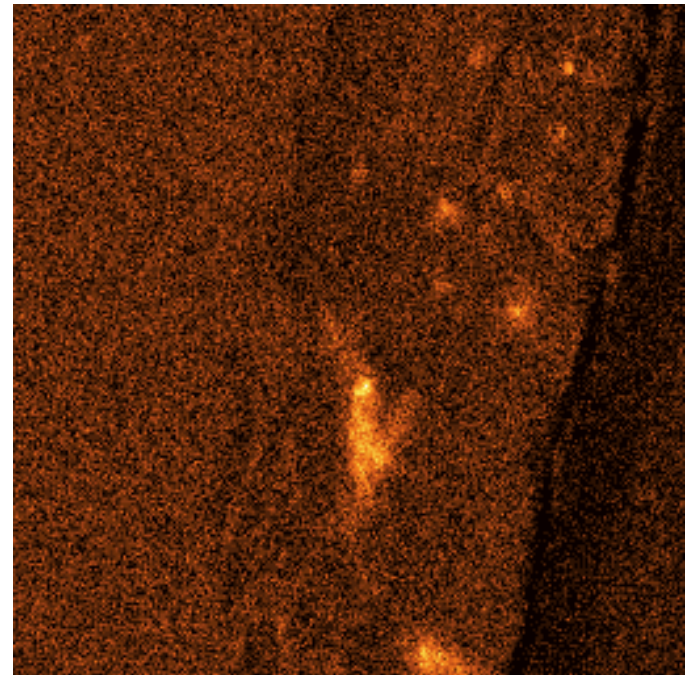


Elemental Composition

Elemental Mapping using Electron Microprobe Analysis



Corrosion
Deposits



Arsenic distribution

Elemental Composition

Cross-sectional Elemental Mapping

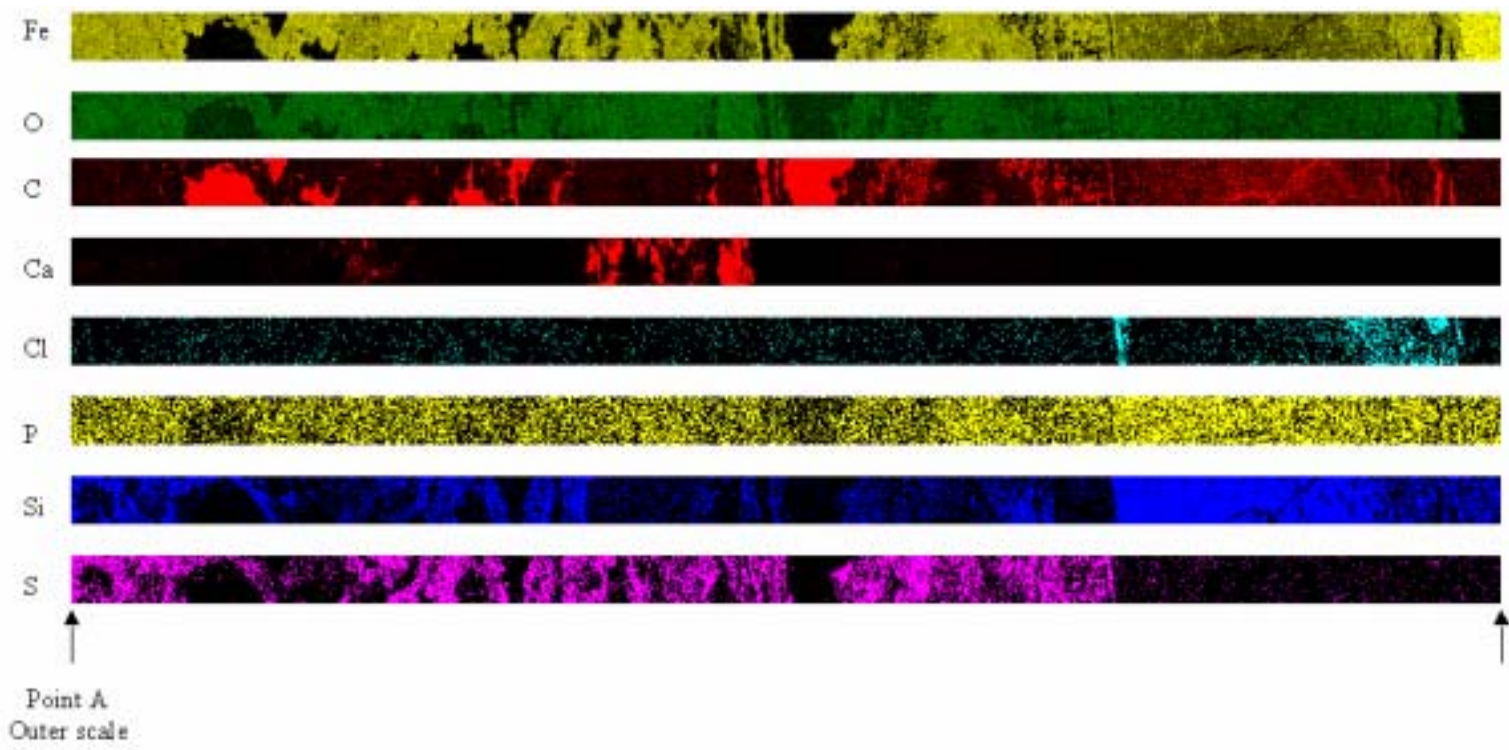


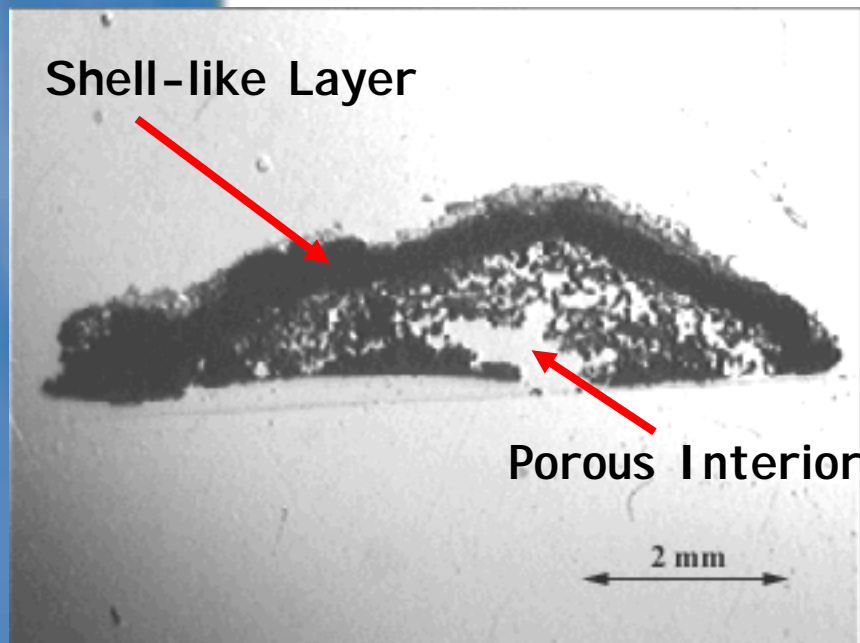
Figure 7. Elemental mapping of cross section of sample DL00046 by electron microprobe (240X).

Tubercle Structure



Typical Scale Structure and Composition

- Corrosion scales are *porous deposits* with a hard *shell-like layer*
- *Reservoir of Fe(II)* ions exists in the scale interior
- **Composition**
 - *Shell-like layer*: Magnetite (Fe_3O_4) and goethite ($\alpha\text{-FeOOH}$)
 - *Porous Interior*: Mostly Fe(II) compounds, green rusts (possible), and ???
 - Fe(III) compounds present only in the top layers. Also other solids.



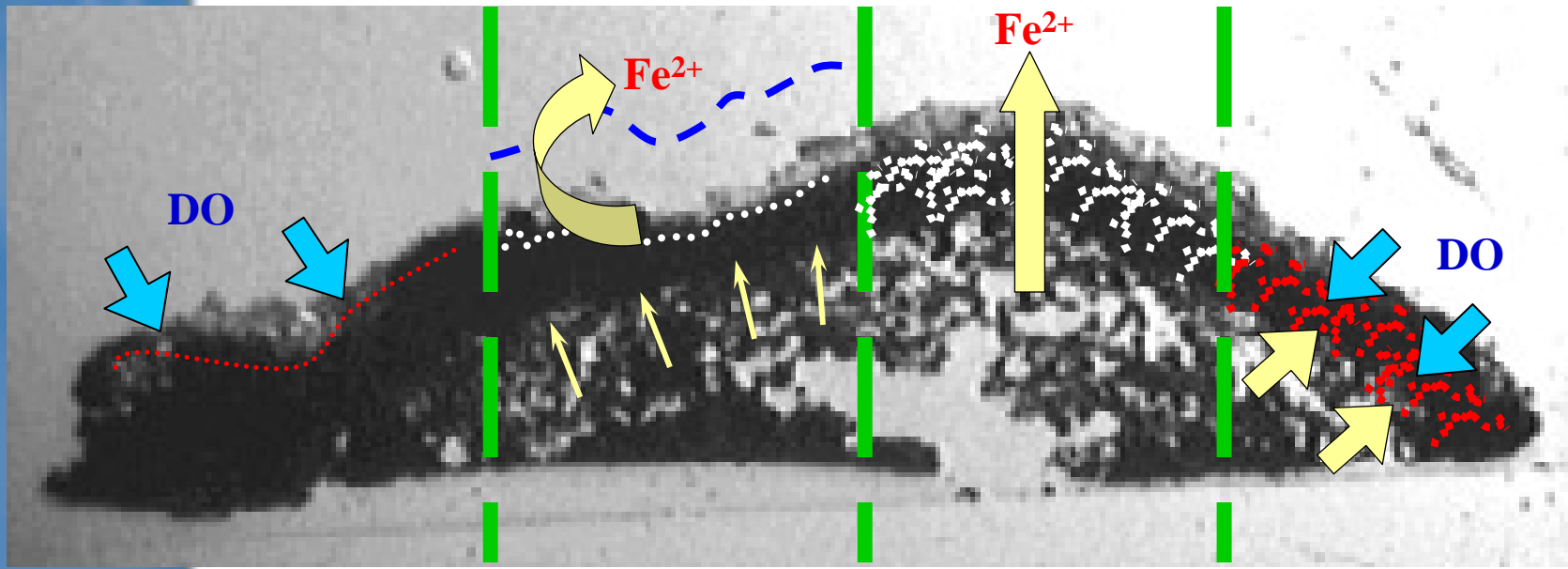
Iron Release from Corrosion Scales

Flowing Water
with oxidants

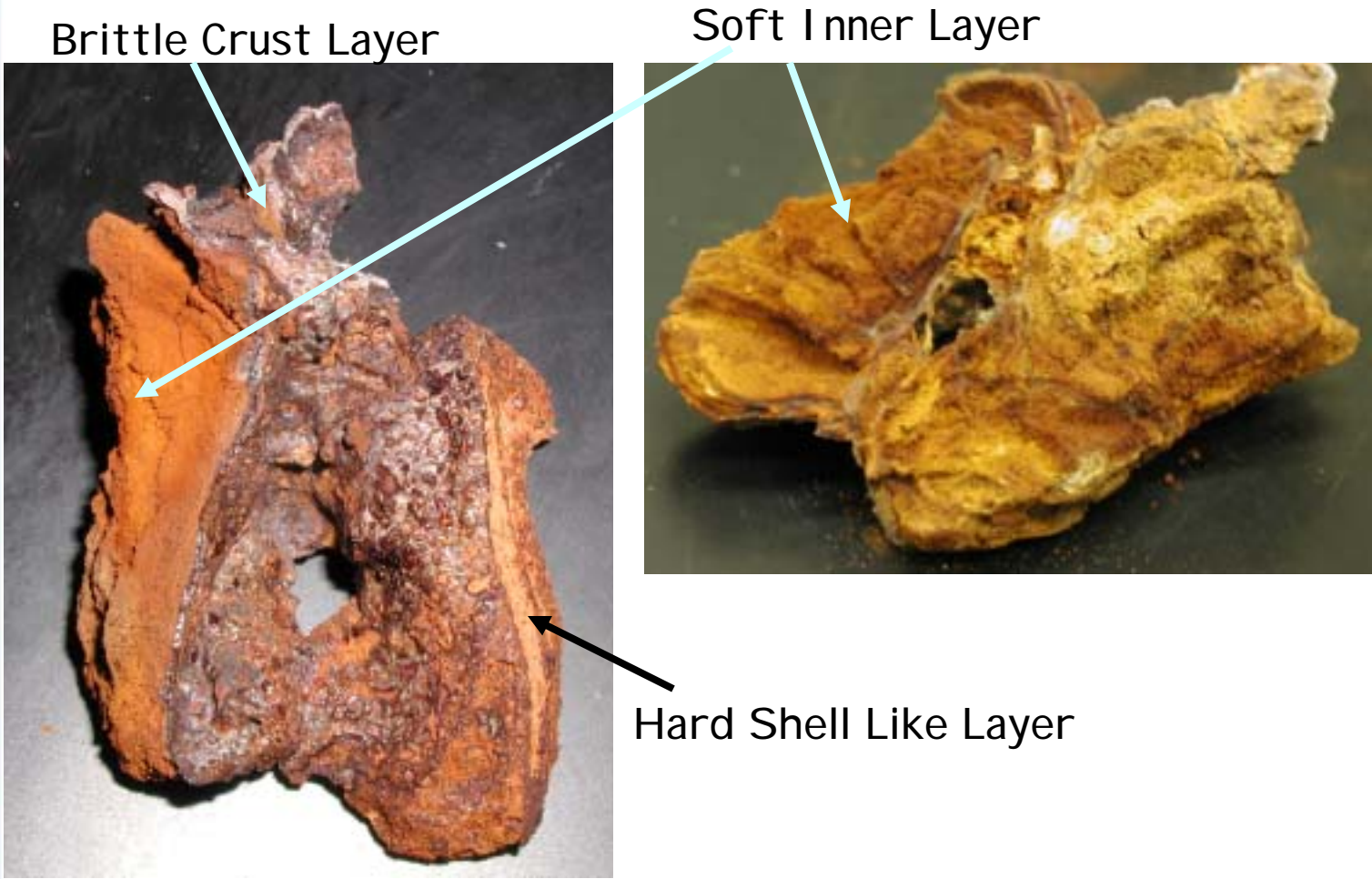
Stagnant Water
with oxidants
"Anoxic layer"

Prolonged
Stagnation

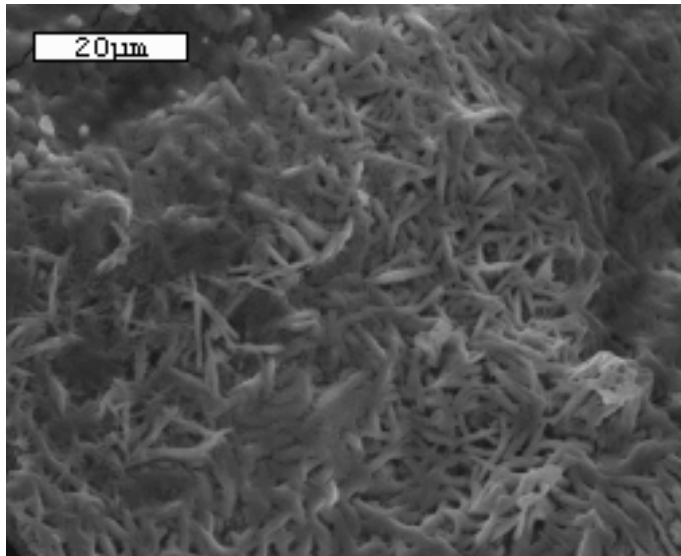
Oxidant supply
restored



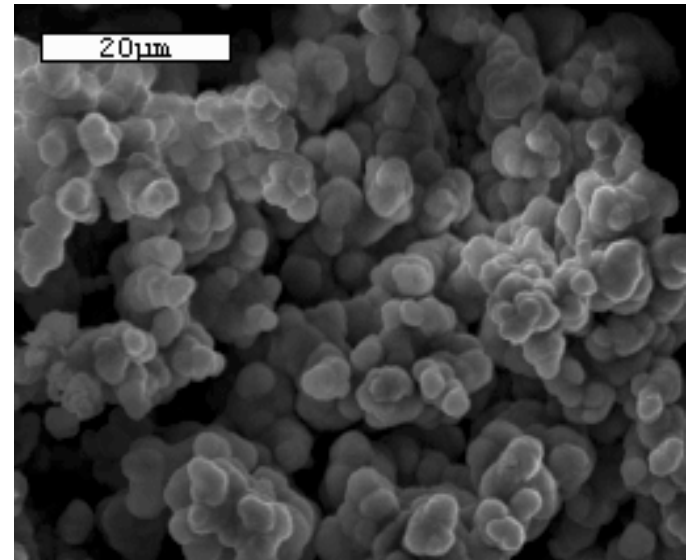
Tubercle Structure



Tubercle Structure



Hard outer scale layer



Soft inner layer

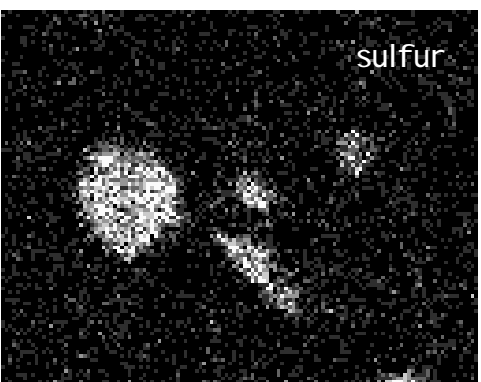
The Role of Microorganisms? SRBs



Iron drinking water main



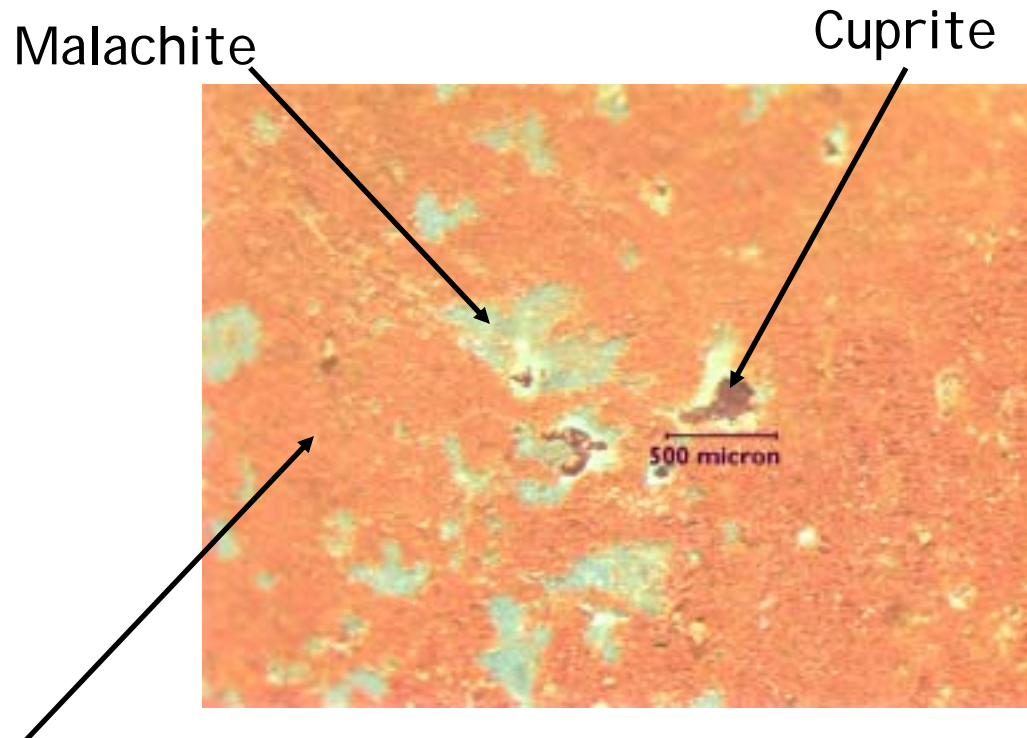
Scale structure



sulfur

SEM micrographs and
Elemental mapping

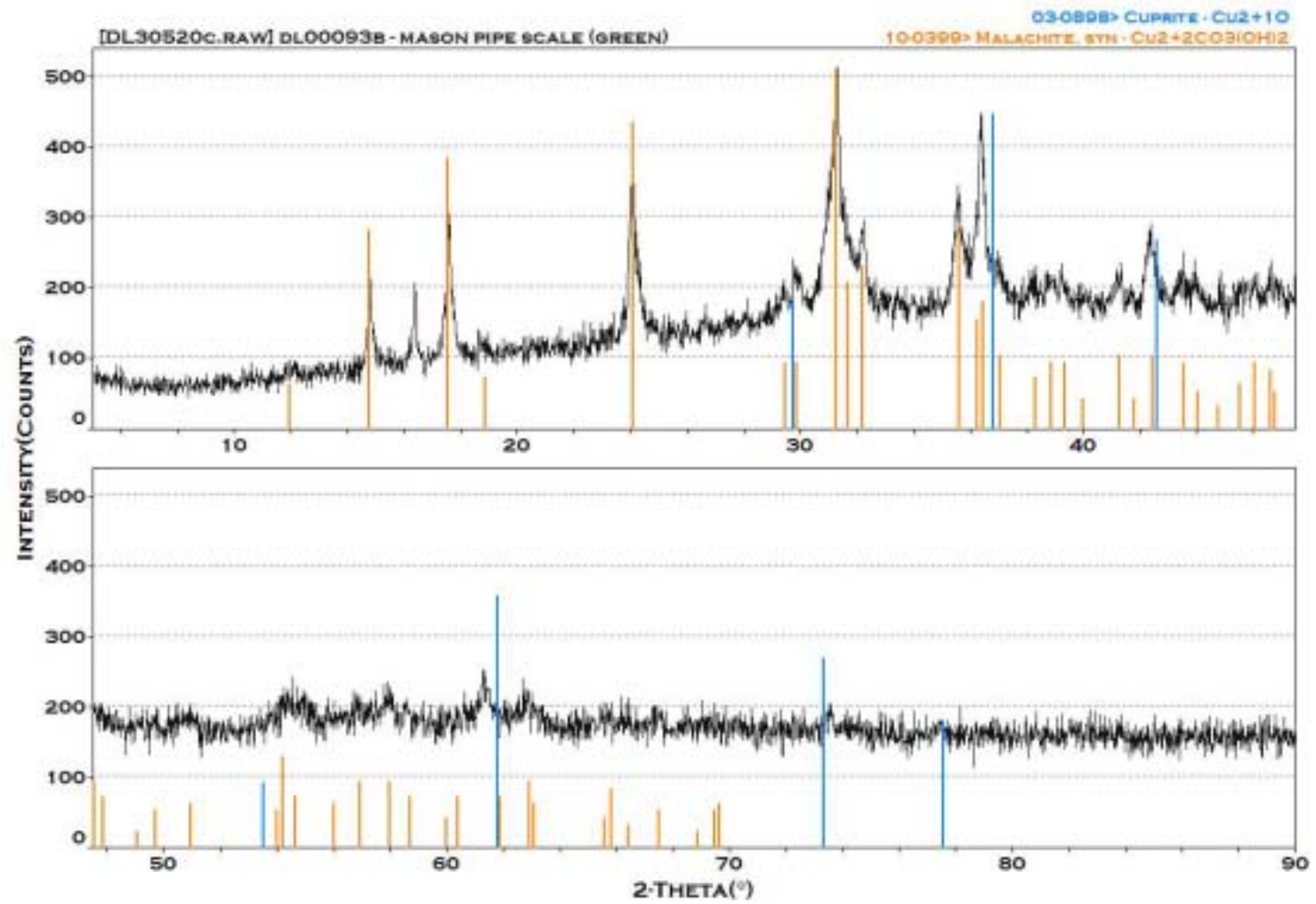
Corrosion of Copper



Possible iron precipitate from distribution system

Building a
scientific
foundation
for sound
environmental
decisions

Copper Corrosion Products

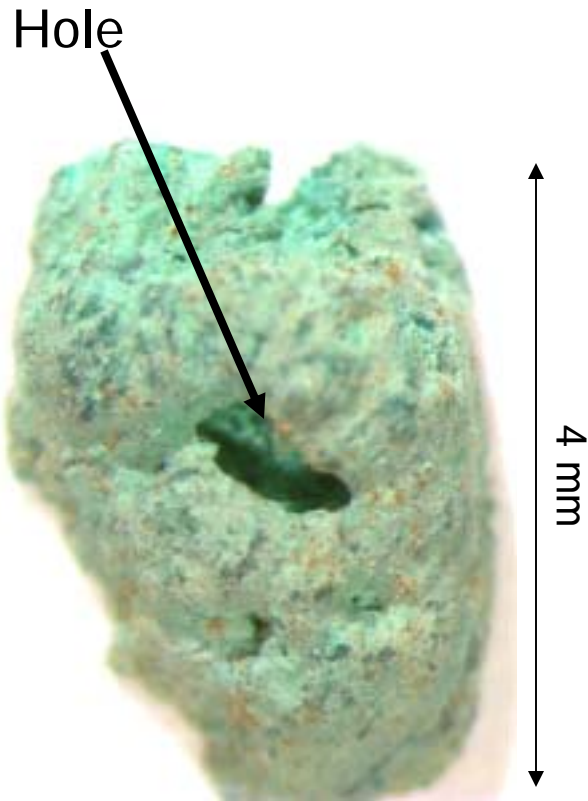


Copper Pitting



Pits are found beneath corrosion deposits

Copper Pitting



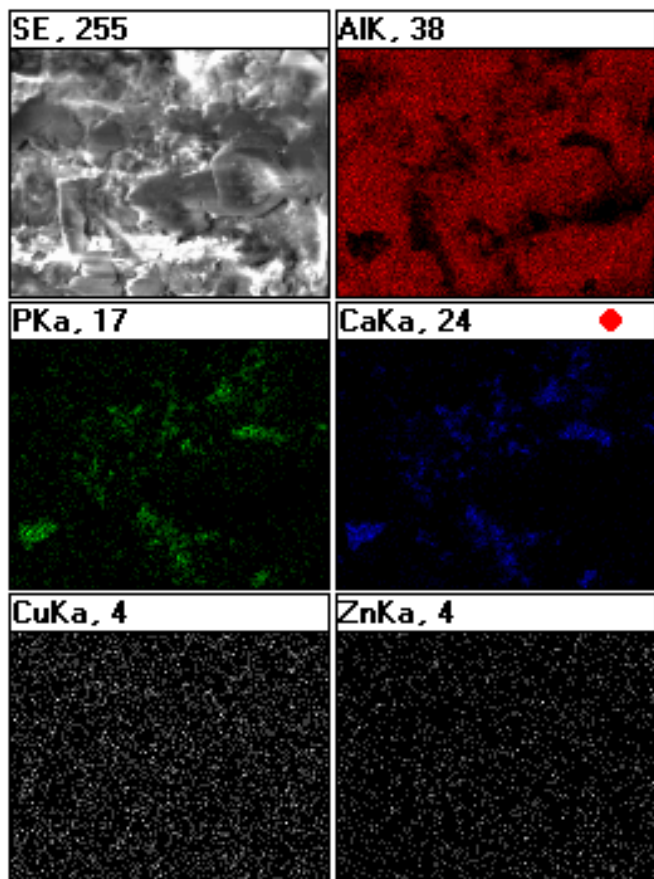
Copper Corrosion
Deposit



Pit Beneath Corrosion
Deposit

Identification of Suspended Solids

SEM/EDS Elemental Mapping



Conclusions

- Analysis of distribution system solids should be approached using multiple techniques
- Elemental composition and trace metal contamination in DS solid can be determined
- Corrosion theory can be tested or established
- Metal solubility predictions can be confirmed and models can be improved